Influence of temperature stability on sensing properties of SAW NO$_x$ sensor

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Received 8 November 2006; accepted 28 February 2008

This paper presents the study on the influence of temperature stability of SAW sensor on the sensing properties for NO$_x$ gas. Temperature stable SAW sensor based on 128° YX LiNbO$_3$ single crystal was developed after integrating with rf sputtered amorphous TeO$_2$ thin film that act as a temperature compensation over-layer. TeO$_2$ thin films possessing negative temperature coefficient of delay (TCD) also served as a sensing layer for the detection of NO$_x$ gas. Temperature stable SAW devices are essentially desired to avoid the problem of false alarm, besides the enhancement in the sensitivity and reliability.

Surface acoustic wave (SAW) gas sensors have been in commercial use in the field of environmental protection, automotive emission control, medical applications and process engineering due to small size, less expensive, high sensitivity and remote sensing. The basic principle of the SAW gas sensor is the change in the mass and electrical properties of a specifically chosen sensing over-layer due to impinging gas/chemical molecules through physical absorption, adsorption, or other mechanism and thereby influence signal flow of the device. Most of the commercially used SAW devices are based on 128° YX LiNbO$_3$ single crystal, a well-known substrate possessing high-electromechanical-coupling coefficient, but its high temperature coefficient of delay (TCD) = 75-100 ppm/°C leads to reliability problem. The uncompensated temperature drifts in the SAW sensor tends to interfere with its actual sensing characteristics. In the present study the influence of temperature stability of the SAW device on the sensing characteristics for NO$_x$ gas has been studied.

**Experimental Procedure**

Tellurium oxide (TeO$_2$) thin films were deposited by rf sputtering under reactive ambient (75% Ar + 25% O$_2$) using optimized processing conditions reported elsewhere. TeO$_2$ films of different thickness were grown over SAW device fabricated on 128° Y-X LiNbO$_3$ single crystal operating at 36 MHz. Periodicity ($\lambda$) of the aluminum inter-digital transducers (IDTs) was 106 µm. Thickness of the films was measured using a surface profiler (Dektak IIA). X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) studies are used to characterize the film. The SAW propagation and TCD characteristics of layered structure were studied using Agilent (8712ES) RF network analyzer. Sensing NO$_x$ gas characteristics of SAW layered structure was investigated by keeping the device in specially designed gas test rig.

**Results and Discussion**

Tellurium oxide (TeO$_2$) film grown on corning glass substrate was found to be uniform, transparent and strongly adherent. No discernible crystallization was observed in the XRD spectra indicating the formation of amorphous films. The FTIR spectra of film deposited on KBr pellets show strong absorption band at ~630 cm$^{-1}$ corresponding to Te-O bond and are well within the range reported for TeO$_2$ bulk and thin films. Density (~5766 kgm$^{-3}$) of the film using weight gain and thickness measurement was found to be slightly less in comparison to 5900 kgm$^{-3}$ reported for TeO$_2$ single crystal.

**SAW layered structure**

The operating frequency of LiNbO$_3$ SAW device shifts from 36.88 MHz to 36.69 MHz with increase in ambient temperature from 25°C to 95°C. The observed shift ($\Delta F$) reduces after deposition of TeO$_2$ thin films of increasing thickness over SAW device. TCD of device was determined from the slope of plot between the $\Delta F$ versus ambient temperature. The
variation of TCD as a function of normalized thickness of overlayer for TeO$_2$/LiNbO$_3$ SAW devices is shown in Fig. 1. TCD of layered device decreases with increase in overlayer thickness and ultimately attains a zero value resulting in a temperature stable device.

Three SAW device structures (Fig. 2 a, b and c) were considered for further study: (i) uncoated LiNbO$_3$ device (device A; TCD = 75 ppm °C$^{-1}$), (ii) TeO$_2$ (0.021$\lambda$)/IDT/LiNbO$_3$ (device B; TCD = 35 ppm °C$^{-1}$), and (iii) TeO$_2$(0.042$\lambda$)/LiNbO$_3$ (device C; TCD = 0). Figure 3 shows the change in center frequency of devices A, B and C as a function of ambient temperature. The increase in temperature of device B exhibit a small shift in frequency in comparison to device A (Fig. 3), and is due to its partial temperature compensation after integrating with 2.25 µm thick TeO$_2$ over layer. Whereas device C (TeO$_2$ thickness ~ 4.5 µm) exhibits $\Delta F = 0$.

NO$_x$ gas sensing response

A downshift in center frequency ($\Delta F$) by 58 kHz and 20 kHz was observed for device A and B respectively with NO$_x$ gas (0.01 vol%), whereas an upshift was seen for device C. The effect of temperature instability of device A and B towards $\Delta F$ leads to such inconsistent behaviour under NO$_x$ gas. Figure 4 is the plot of $\Delta F$ versus concentration of NO$_x$ gas for all devices. The response of device A was independent of NO$_x$ concentration, whereas a continuous upshift in frequency of device C was seen with increasing NO$_x$ concentration (Fig. 4). The sensing response of device B could be divided into two region (Fig. 4); (i) Downshift frequency under low concentration (<0.045 vol%), and (ii) Upshift frequency under high concentration (> 0.045 vol%). The shift $\Delta F$ can be interpreted as $\Delta F_1 + \Delta F_2$, where $\Delta F_1$ is the contribution due to temperature instability of device, and $\Delta F_2$ is due to interaction of gas with...
sensing layer. Since device A exhibits $\Delta F = -58$ kHz for all concentration, the value of $\Delta F_1 = -58$ kHz and $\Delta F_2 = 0$. Device C with zero TCD shows upshift under NO$_x$, therefore $\Delta F_1 = 0$ and $\Delta F = \Delta F_2 > 0$. Device B is partly temperature compensated, therefore $\Delta F$ have the contribution from both $\Delta F_1$ and $\Delta F_2$, and $\Delta F < \Delta F_2$ due to negative contribution of $\Delta F_1$. It may be noted that the difference of temperature between devices (~23°C) and NO$_x$ gas (~55°C) is about 20°C. The observed $\Delta F = 0$ for device B with 0.045 vol% NO$_x$ indicate that the contribution of $\Delta F_1$ due to increase in temperature by 20°C is cancelled by $\Delta F_2$ due to actual sensing of NO$_x$ by TeO$_2$. Considering TCD = 35 ppm °C$^{-1}$ for device B (Fig. 3), the estimated value of $\Delta F_1$ is $-24$ kHz, and the value of $\Delta F_2 = +24$ kHz for 0.045 vol% instead of observed value of zero ($\Delta F = 0$).

It may be noted from Fig. 4 that $\Delta F_2$ increases with increase in NO$_x$ concentration (device C), whereas $\Delta F_1$ was independent of concentration (device A). Thus, $\Delta F$ of device B was downshift for low concentration (< 0.045 vol%) and upshift at high concentration (> 0.045 vol%) of NO$_x$. The actual shift ($\Delta F_2$) for device B was also obtained after suppressing the contribution of temperature instability of device, and is plotted in Fig. 4. It may be noted (Fig. 4) that actual shift ($\Delta F_2$) for device B was upshift in the entire range of NO$_x$ concentration, and is in agreement to the response of device C. Therefore, the interaction of NO$_x$ with TeO$_2$ sensing layer shift the center frequency of SAW device towards higher frequency. The low sensitivity ($\Delta F_2$) observed for device B in comparison to device C for a given NO$_x$ concentration (Fig. 4) is due to the reduction in thickness of TeO$_2$ sensing layer, from 0.042$\lambda$ to 0.021$\lambda$ in device B. Therefore, the temperature stable SAW devices with TCD = 0 are essentially required for gas sensor application to make the output free from any change in ambient temperature and to avoid the false alarm problem.

Conclusions
The temperature stability of SAW device plays a crucial role in designing an efficient and reliable gas sensor. The rf sputtered TeO$_2$ thin film act as a sensing as well as a temperature compensation layer in TeO$_2$/128°YX LiNbO$_3$ layered SAW structure. The zero TCD device structure restricts the temperature induced interference towards the actual sensing response characteristics, and enhanced response for NO$_x$ gas is obtained.

Acknowledgements
Authors are thankful to DRDO and DST, Govt. of India for financial support to carry out this work.

References